AD A

INVESTIGATION OF ON-VEHICLE FUEL CATALYST SYSTEM

INTERIM REPORT TFLRF No. 375

by

Edwin A. Frame
U.S. Army TARDEC Fuels and Lubricants Research Facility SwRI®
Southwest Research Institute®

San Antonio, TX

for

U.S. Army TARDEC Petroleum and Water Business Area Warren, MI

Contract No. DAAE-07-99-C-L053 (WD16) SwRI Project No. 03.03227.16

Approved for public release; distribution unlimited

September 2004

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Edwin C. Owens

Director

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REPORT DOCUMENTATION PAGE

Form Approved OMB No. 0704-0188

Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instruction, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.

1. AGENCY USE ONLY	2. REPORT DATE September 2004	3. REPORT TYPE AND DATES COVERED Interim, September 2002 – September 2004		
4. TITLE AND SUBTITLE Investigation of On-Vehicle Fuel Catalyst 6. AUTHOR(S) Frame, E.A.as M.	5. FUNDING NUMBERS DAAK70-92-C-0059; WD 16			
7. PERFORMING ORGANIZATION NAM U.S. Army TARDEC Fuels and Lubricant Southwest Research Institute P.O. Drawer 28510 San Antonio, Texas 78228-0510	8. PERFORMING ORGANIZATION REPORT NUMBER TFLRF No. 375			
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army TACOM U.S. Army TARDEC Petroleum and Water Business Area Warren, MI 48397-5000			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited			12b. DISTRIBUTION CODE	

13. ABSTRACT (Maximum 200 words)

On-vehicle pre-combustion fuel catalytic devices have the potential to improve fuel performance in the areas of decreased fuel consumption, reduced exhaust emissions and restored fuel properties. SwRI/TFLRF subcontracted with Advanced Power Systems, Incorporated (APSI) to develop enhanced fuel pre-combustion catalyst systems for use with diesel fuel and JP-8 fuel. APSI developed and delivered three fuel pre-combustion catalyst systems. The systems will be evaluated in the future by TACOM.

SwRI/TFLRF evaluated a baseline fuel catalyst system for its effect on fuel economy and fuel property changes. Under the conditions tested, no fuel economy benefit was observed for the baseline device. The stability of aged diesel fuel was not improved by flowing fuel through the baseline device.

14. SUBJECT TERMS Diesel Fuel C	ombustion JP-8 Fuel Catalyst System		15. NUMBER OF PAGES
			16. PRICE CODE
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89) Prescribed by ANSI Std. Z39-18

FOREWARD/ACKNOWLEDGEMENTS

This work was performed by the U.S. Army TARDEC Fuels and Lubricants Research Facility (TFLRF) located at Southwest Research Institute (SwRI), San Antonio, Texas, during the period September 2002 through September 2004 under Contract No. DAAE-07-99-C-L053. The project was administered by the U.S. Army Tank-Automotive RD&E Center, Petroleum and Water Business Area, Warren, Michigan. Mr. Luis Villahermosa (AMSRD-TAR) served as the TARDEC contracting officer's technical representative.

The author would like to acknowledge the contribution of the TFLRF technical support staff along with the administrative and report-processing support provided by Linda De Salme.

BACKGROUND

On-Vehicle pre-combustion catalytic treatment of fuel has the potential to improve fuel performance in the areas of combustion, exhaust emissions and fuel economy. In addition, fuel catalyst treatment may resuscitate fuels that have become oxidized or contaminated with bacterial microorganisms.

EXECUTIVE SUMMARY

On-vehicle pre-combustion fuel catalytic devices have the potential to improve fuel performance. Manufacturers of these devices claim benefits are possible in fuel economy, reduced exhaust emissions and restored fuel properties. SwRI/TFLRF subcontracted with Advanced Power Systems, Incorporated (APSI) to develop an enhanced fuel pre-combustion catalyst system for use with diesel fuel and JP-8 fuel. APSI developed and delivered three fuel pre-combustion catalyst systems. The systems will be used in future TACOM evaluations.

SwRI/TFLRF evaluated a baseline fuel catalyst system for its effect on fuel economy and fuel property changes. Under the conditions tested, no fuel economy benefit was observed for the baseline device. The stability of aged diesel fuel was not improved by flowing fuel through the baseline device.

Evaluation of the enhanced devices will be conducted in another program.

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1. OBJECTIVE AND APPROACH

The objective of this project was to evaluate the effectiveness of an on-vehicle fuel catalyst system when used with diesel fuel and kerosene type fuels. Potential benefits include improved fuel economy, reduced exhaust emissions, increased power, and elimination of the need for some fuel additives. A subcontract was issued to Advanced Power Systems International, Inc. (APSI) to enhance their research effort to develop a fuel catalyst system for use with diesel and JP-8 fuels. The effect on fuel properties of exposure to the baseline fuel catalyst system was determined. In addition, the fuel catalyst system was evaluated for its effect on the fuel economy of a small diesel engine.

2. EVALUATION OF FUEL CATALYST SYSTEM

2.1 Preparation of Aged JP-8 and DF-2.

APSI requested that TFLRF provide them with "degraded" fuel that could be used in their experimental efforts. APSI was initially provided with samples of JP-8 (AL-26936) and DF-2 (EPA Low Sulfur Certification Fuel, AL-26939) that had not been aged/degraded. These fuels were for baseline tests.

A drum of the JP-8 fuel (AL-26936) and a drum of the DF-2 fuel (AL-26939) were aged/degraded at TFLRF prior to shipment to APSI. The fuels were aged at 65-70 C with air bubbled in the fuel. Figure 1 shows the fuel aging setup. The JP-8 was aged approximately six months. Determining the JFTOT breakpoint (ASTM D3241) monitored JP-8 degradation. The new JP-8 has a breakpoint of 285 C, and the aging did not substantially reduce the breakpoint. A small quantity of nitrogen material was added to the fuel to expedite aging and degradation of the JP-8. The tests with added nitrogen compounds (dimethyl pyrolle, and then indole) did not have the expected effects. The fuel did exhibit some discoloration. It was shipped to APSI.



Figure 1. Fuel Aging Set up

The diesel fuel degradation was monitored by ASTM D6468. This fuel was aged approximately 3.5 months. The reflectance rating was decreased from an initial value of 85 and reached a stable value of 68. As shown in Figure 2, this fuel was supplied to APSI. Successful application of the fuel catalyst should return the rating to at least 80 (the minimum for premium diesel fuel).

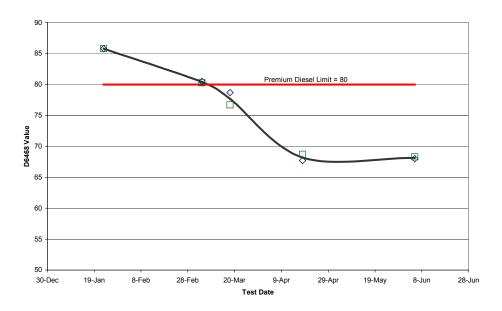


Figure 2. History of Diesel Thermal Stability

2.2 Evaluation of Baseline Fuel Catalyst System

TFLRF received baseline, in-line canister fuel catalyst systems from APSI. The catalyst was FFC-A, a catalyst sold commercially by APSI for use in gasoline fueled engines. The baseline device (FFC) was evaluated for its effect on fuel properties and on fuel consumption in a small diesel engine.

2.2.1 Effect on Fuel Properties

A fuel flow through apparatus was set up as shown in Figure 3. The fuel was pulled from a drum and entered a heating element where it was immediately heated to the test temperature. Fuel temperature was monitored both into and after the baseline FFC. The fuel was returned to the drum after passing through the FFC. In the test procedure, the FFC was conditioned for 72 hours by re-circulating the 20-gallon fuel sample at a flow rate of 15 gallons per hour. Fuel temperature into the FFC was controlled at 100F. Fuel temperature after the FFC was steady at 100F. After 72 hours, a fuel sample was taken for analysis. The same aged fuel remained in the flow system, and the fuel temperature into the FFC was raised to 130°F. Fuel flowed at 15 gallons per hour for an additional 72 hours, and then another sample was taken for analysis. Aged JP-8 (AL-27003) and aged diesel fuel (AL-27006) were tested using this procedure. A new baseline FFC was used for each fuel type. The fuel samples that were collected were analyzed for changes in properties and performance as shown in Table 1 (JP-8) and Table 2 (Diesel). Fuel flow through the FFC did not add elemental contamination. JP-8 and diesel fuel lubricity performance remained constant. All changes in Cetane number by IQT method were within test repeatability. The diesel fuel stability as determined by ASTM D6468 was not improved. Thus, the FFC did not demonstrate the ability to resuscitate diesel fuel.



Figure 3. Fuel Flow through Apparatus

The following compositional changes were observed for the diesel fuel after re-circulation through the FFC:

- Reduction in Total Aromatic content at the higher test temperature and longer flow time.
- Reductions in PNA at both lower and higher test temperatures.

For the JP-8, the following composition changes were observed:

- Reduction in aromatics
- Reduction in carbon content after 72 hours at 130°F.

Additional investigation would be needed to isolate and determine the exact cause of these fuel composition changes.

Table 1. FFC Testing with JP-8 Fuel						
Description			AL-27003 CL04-0183 aged JP-8	CL04-0078 72 hrs, 100F	CL04-0079 72 hrs, 130F	CL04-0080 144 hrs 130F
Property	Test Method	Repeatability				
Density, g/cc	D4052		0.7948	0.7949	0.7952	0.7953
Kvis 40C, cSt	D445		1.21	1.21	1.22	1.23
Carbon, w%	D5291	+/- 1	85.81	85.6	84.44	84.96
Hydrogen, w%	D5291		14.28	15.24	14.77	14.94
Total Aromatics, w%	D5186	+/- 0.3	19.33	18.98	18.91	19.01
Mono-Aromatics, w%	D5186		16.58	16.3	16.21	16.13
PNA, w%	D5186	+/- 0.2	2.75	2.69	2.7	2.88
Lubricity 60C, microns	D6079	+/- 80	640	640	600	655
Lubricity, BOCLE, mm	D5001	+/- 0.03	0.48	0.5	0.49	0.51
Sulfur, w%	D5185		0.009	0.01	0.01	0.009
Cetane Number,+/- 2	IQT	+/- 2	47.7	48	48.5	48.5

Table 2. FFC Testing with Diesel Fuel						
Description			AL-27006 CL04-0184 aged diesel	CL04-0045 72 hrs, 100F	CL04-0046 72 hrs, 130F	CL04-0047 144 hrs 130F
Property	Test Method	Repeatability				
Density, g/cc	D4052		0.8473	0.8473	0.8474	0.8474
Kvis 40C, cSt	D445		2.76	2.77	2.78	2.78
Pour point, C	D5949		-21	-21	-21	-21
Flash Point, C	D93		93	95	95	95
Hydrocarbon type	D1319					
Aromatics, v%		+/- 1.4	31	31.3	29.6	28.3
Saturates, v%		+/- 1.7	68	67.6	68.9	70.4
Olefins, v%		+/- 0.4	1	1.1	1.5	1.3
Total Aromatics, w%	D5186	+/- 0.3	31.64	31.29	30.64	30.23
Mono-aromatics, w%	D5186		21.24	21.34	21.58	21.53
PNA, w%	D5186	+/- 0.2	10.4	9.95	9.06	8.7
Carbon, w%	D5291	+/- 1	86.46	86.72	86.67	86.6
Hydrogen, w%	D5291		13.02	12.96	13.19	13.14
Lubricity 60C, microns	D6079	+/- 80	375	375	310	330
Stability, % reflectance	D6468	+/- 7.5	70.9	70.6	71.7	70.4
Copper Corrosion	D130		1A	1A	1A	1A
Sulfur	D5185		0.036	0.036	0.036	0.036
Cetane Number	IQT	+/- 2	46.3	47.1	47.4	47.4

2.2.2 Fuel Consumption Effects

The effect of the baseline FFC on fuel consumption was determined using the 3-cylinder Kubota diesel engine/generator set following a fuel consumption test procedure developed by TFLRF and used in the evaluation of engine lubricant effects on fuel consumption. Figure 4 shows the Kubota engine test installation. In this procedure, total fuel consumption is measured with the engine operating at steady state. The test duration is four hours. In previous work, fuel consumption changes of 2 to 3 % have been determined with excellent repeatability. A fresh batch of Army reference oil (SAE 15W40) was used for each fuel consumption test. The fuel catalyst system was installed in the fuel inlet line. It was conditioned for 72 hours while operating the engine at fuel consumption test conditions where the fuel into the FFC was 74°F. The test fuel for all fuel consumption tests was Jet-A additized with MIL-PRF-25017 corrosion inhibitor/lubricity improver. The average fuel consumption for all previous tests with this Army reference oil was 19.46 lbs. Six fuel consumption tests were conducted: three without the FFC and three with the fuel flowing through the FFC. The test matrix and results are shown in Table 3.

Table 3. Fuel Consumption Tests in Kubota Diesel Engine				
	Fuel Consumption, lbs			
Date	Without FFC	With FFC		
2-4-04	19.48	X		
2-5	X	19.51		
2-6	19.53	X		
2-9	X	19.53		
2-10	19.46	X		
2-11	X	19.52		
Average	19.49	19.52		

No fuel consumption improvement was observed with the baseline FFC in the fuel line.



Figure 4. Kubota Engine Installation

3. CONCLUSIONS

The following conclusions are made based on the evaluation of the baseline FFC:

- Under the conditions tested, using the three-cylinder Kubota diesel engine, no fuel consumption improvement was observed with the baseline FFC in the fuel line.
- Diesel fuel stability as determined by ASTM D 6468 was not improved by re-circulating aged diesel fuel through the baseline FFC.
- Some minor compositional changes were observed in aged diesel fuel PNA content after fuel recirculation through the baseline FFC. Higher fuel temperature and extended re-circulation time resulted in lower PNA content of the aged diesel fuel.
- For aged JP-8 there was a slight reduction in fuel aromatic content after re-circulation through the baseline FFC.
- Additional investigation would be needed to isolate and determine the exact cause of these fuel compositional changes.

4. SWRI SUBCONTRACT WITH APSI

SwRI issued a subcontract (3994750) to APSI, Inc. The purpose of the subcontract was to "extend the capability of the patented pre-combustion Fitch Fuel Catalyst (FFC) to diesel and JP-8 fuel". APSI completed all subcontract work with the delivery of one baseline and three other revised fuel catalyst systems. The three revised fuel catalyst systems were visually inspected by TFLRF and then returned, on May 7, 2004, to APSI for use in other TACOM projects.

The tree revised fuel catalyst systems were designated:

- FFC-A-F8T, AL-26957
- FFC-C, AL-27052
- FFC-C + M, AL-27053

Appendix A contains the final subcontract report from APSI.

5. RECOMMENDATIONS

The following recommendations are made:

- The revised fuel catalyst devices that APSI prepared under subcontract to SwRI should be evaluated for their effects on vehicle fuel economy, exhaust emissions, and fuel properties.
- If the revised fuel catalyst devices show beneficial performance in laboratory evaluation tests, a well-controlled pilot field demonstration should be conducted using Military ground wheeled vehicles (non-tactical vehicles).

APPENDIX A

APSI REPORT OF FUEL CATALYST RESEARCH AND EVALATION FOR SWRI
CONTRACT NO. 3994750
VOLUME 1 AND VOLUME 2

ADVANCED POWER SYSTEMS INTERNATIONAL INC.

558 Lime Rock Road Lakeville, CT 06039 tel 860-435-2525 fax 860-435-2424 Website – www.fitchfuelcatalyst.com

February 2004

RE: SwRI Contract number 399475O – Advanced Power Systems International, Inc.

APSI's final submittal to SwRI for this contract consists of two volumes.

Volume I

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- 8. Monthly Progress Report #8
- 9. Monthly Progress Report # 9
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- 12. Monthly Progress Report #12
- 13. Certification of APSI as to Progress Report 1-12 Completeness and Accuracy
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- 19. Figures 1-11
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- 22. Plans for catalyst scale-up, laboratory testing, and field tests

Volume 1 TAB 1.

1ST MONTHLY PROGRESS REPORT SWRI CONTRACT 3994750

DEC. 5, 2002

Introduction:

This report covers work performed between the period of November 25, 2002 (the start date of the contract) and December 3, 2002 by Advanced Power Systems International (APSI)...

Accomplishments:

- 27. Contract signed and returned to SWRI
- 28. Negotiations commenced with all subcontractors
- 29. Communication with Ed Frame to ask his help in securing appropriate samples of fuels for testing with catalysts.
- 30. Review initiated of recognized test methodologies for microorganism growth and oxidation in JP-8 and diesel fuel.

Test Data and/or Summary Technical Discussion:

No tests were conducted in this period

Plans for next period:

- 1. Sign contracts with all subcontractors
- 2. Obtain "clean" and "degraded" fuel samples of JP-8 and Diesel from SWRI (approximately 5 gallons) for use at UCONN during program. Fuel will be from "batches" to be retained at SWRI for testing there later in program
- 3. Establish UCONN laboratory test procedures for comparing treated (with catalyst) and untreated fuel.
- 4. Establish ASTM (or equivalent) test methodologies to be used to compare treated or untreated fuels
- 5. Initiate testing at UCONN

William Lueckel

APSI Program Manager Volume 1 TAB 2.

2nd MONTHLY PROGRESS REPORT

SWRI CONTRACT 3994750

JAN. 5, 2002

Introduction:

This report covers work performed between the period of December 3, 2003 and January 3, 2003 by Advanced Power Systems International (APSI)

Accomplishments:

- 1) Contracts signed with all Subcontractors and Consultants
- 2) Work Plan for January and February 2003 prepared
- 3) Obtained agreement of SWRI to provide 5 gallons each of four fuels: Clean Diesel, Degraded Diesel, Clean JP-8, and Degraded JP-8. Fuel to be used at UCONN in test program
- 4) Established UCONN laboratory test procedures for comparing treated (with catalyst) and untreated fuel.

Test Data and or Summary Technical <u>Discussion</u>:

Copy of January / February Work Plan Attached.

Plans for next period:

- 1) Receive "clean" and "degraded" fuel samples of JP-8 and Diesel from SWRI (approximately 5 gallons) for use at UCONN during program. Fuel will be from "batches" to be retained at SWRI for testing there later in program
- 2) Initiate Testing at UCONN, Validate test methodologies with untreated JP-8 and Diesel Fuels
- 3) Obtain copy of engine owner manual from SWPI for engine to be used in testing treated fuel at SWRI

William Lueckel

APSI Program Manager

Volume 1 TAB 3.

3rd MONTHLY PROGRESS REPORT SWRI CONTRACT 3394750

FEB 6, 2003

Introduction:

This report covers work performed between the period of January 3, 2003 and February 3, 2003 by Advanced Power Systems International (APSI).

Accomplishments:

- a. Received 5 gallons each of "clean" Diesel and JP-8 fuel from SWRI.
- b. Activity per Work Plan for January and February 2003 commenced.
- c. Initiated UCONN laboratory test procedures. NMR and Liqui-Cult tests for untreated Diesel and JP-8 fuel completed.
- d. Located laboratory in New Haven, CT able to perform fuel ASTM oxidation stability and other tests per work plan.
- e. Developed layout and began equipment procurement for experimental apparatus to flow fuels through various catalyst media.

Test Data and/or Summary Technical Discussion:

- 1) Data from Liqui-Cult and NMR tests available upon request.
- 2) Liqui-Cult test showed no microorganisms present in either fuel (expected result)
- 3) NMR test established baseline data for untreated JP-8 and diesel fuel (expected result)

Plans for next period:

- 1) To remain on plan we must receive "degraded" fuel samples of JP-8 and Diesel from SWRI (approximately 5 gallons) within the next two weeks.
- 2) Determine if Saybolt can perform Karl Fisher tests on JP-8 and Diesel fuel. If not locate alternate source.
- 3) Initiate FTIR tests at UCONN on untreated fuels as son as new supply of fluorinated solvents arrived (on order)
- 4) Awaiting copy of engine owner manual from SWRI for engine to be used in testing treated fuel at SWRI

William Lueckel

APSI Program Manager

MEMO

To: Bill Lueckel From: Al Berlin Date: March 2, 2003

Subject: Laboratory Report as an Addendum to Your Feb. 2003 Report to SWRI

Please append this memo describing laboratory work completed at the University of Connecticut, Chemistry Department, at Storrs, in Jan. and Feb. 2003.

Fresh non-aged JP-8 Jet Fuel and Fresh non-aged diesel fuel were examined by proton NMR before and after treatment at ambient temperatures (ca. 22° C), for 72 hours with the Fitch Fuel Catalyst designated as FFC-A. No molecular changes were observed in the JP-8 or Diesel fuels before and after treatment. However, when the proton NMR spectra of these fuels which contained 0.01% trifluoroacetic acid, were compared with these fuels that contained 0.01% trifluoroacetic acid and were treated with FFC-A for 72 hours at ambient temperatures, molecular changes were observed.

Thus, acids are demonstrated to be co-catalysts with FFC-A in changing the molecular structure of JP-8 and diesel fuels.

Both fresh fuels mentioned in the previous paragraphs were analyzed by the Liqui-Cult test method for bacteria and fungi. No bacteria or fungi could be found in either sample.

FTIR methods were tried on the samples, but the cells available were inadequate to obtain results. We wanted to use this method in a quantitative manner to determine the amount of oxidized groups present (olefins, aldehydes, ketones, etc.). We are working to improve our methodology.

Oxygen stability tests, D873-99, and D5304, and water presence will be measured by Karl Fisher on these samples in March.

Signed, A.J. Berlin, Ph.D., 3-2-03

Volume 1 TAB 4.

4th MONTHLY PROGRESS REPORT SWRI CONTRACT 3994750

MAR 6, 2003

Introduction:

This report covers work performed between the period of February 3, 2003 and March 3, 2003 by Advanced Power Systems International (APSI).

Accomplishments:

- 1) Established Plan for testing fuel samples with Saybolt Laboratories in New Haven, Connecticut. They are able to perform planned oxidation tests on both JP-8 and Diesel fuel. Also Karl Fisher tests
- 2) Performed proton NMR examination of fresh non-aged JP-8 and Diesel fuel received from SWRI last month with and without Fuel Catalyst A. As expected, no changes were observed. Liqui-Cult tests showed no bacteria or fungi were present in either the JP-8 or Diesel fuel as received from SWRI
- 3) When small amounts of acid were added to the fresh JP-8 and Diesel fuel samples (to simulate aged fuel) NMR tests showed molecular changes in fuel treated with Catalyst A. The significance of this is that we suspect acid is present in oxidized fuel and possibly fuel containing bacteria and/or fungi.
- 4) Shipped catalyst A to SWRI as required by contract (contract deliverable).

Test Data and/or Summary Technical Discussion:

- 1) Instructions to UCONN regarding shipping fuel to Saybolt Laboratories-letter included in file
- 2) E-mail letter confirming shipment of catalyst A to SWRI-letter attached
- 3) Laboratory Report by Dr. Berlin-report attached

Plans for next period:

- 1) To remain on plan we must receive "degraded" fuel samples of JP-8 and Diesel from SWRI (approximately 5 gallons) within the next two weeks.
- 2) Initiate testing on fuel at Saybolt Laboratory

3) Initiate FTIR tests at UCONN

William Lueckel

APSI Program Manager

ADDENDUM TO THE FOURTH TACOM PROGRESS REPORT

MEMO

To: Bill Lueckel

From: Al Berlin

Subject: Addendum to March 2003 Progress Report

Date: 4-12-03

In March we treated fresh JP-8 with Fitch Fuel Catalyst (FFC-A) for 72 hours. Two treatments were accomplished, one with no addition to the fuel except the FFC-A, and one with fuel, FFC-A, and traces of trifluoroacetic acid (0.01%). These treatments were followed by examination of the results using proton magnetic resonance (PMR), a variant of nuclear magnetic resonance (NMR).

The PMR spectra of untreated JP-8 and the two treated JP-8 samples, as described above, were compared.

The results show unequivocal molecular changes in both treated samples. Because of the complexity of the JP-8 the exact changes cannot be exactly quantitated, but we know for sure that the changes occur in methylene groups (-CH₂-), and the sample treated with FFC-A and its co-catalyst acid show greater changes than the sample that has no acid added.

We are proceeding with similar experiments with fresh diesel fuel, and we require aged fuels to continue our work.

AJB

5th MONTHLY PROGRESS REPORT

SWRI CONTRACT 3994750

APRIL 10, 2003

Introduction:

This report covers work performed between the period of March 3,2003 and April 7, 2002 by Advanced Systems International (APSI).

Accomplishments:

1) APSI shipped first fuel samples to Saybolt Laboratories to perform test series.

The only test commissioned at this time for samples is Karl Fischer test. Dr. Berlins survey of Saybolt concluded it was not optimal for the battery of ASTM fuels tests this program requires. Alternate laboratories are being interviewed.

Samples to be tested are:

- Fresh JP-8, untreated
- Fresh Diesel, untreated
- Fresh JP-8, treated with commercial Fitch Fuel Catalyst (FFC-A)
- Fresh JP-8, treated with FFC-A and "doped" with trifluoroacetic acid (0.01%) (to simulate oxidized fuel)
- 2) Dr. Berlin and the staff at UCONN synthesized high surface area FFC-formula C. These nanoparticles have about 2x10⁵ greater area in square meters per gram than the present FFC-A, and will allow studies to proceed on the effect of surface area on catalyst activity and durability. This high surface area compares favorably with current generation commercial FFC elements where surface area is ~1.7 cm²/gram for the solid elements.
- 3) Tests performed at UCONN allowed us to observe molecular changes induced by the presence of commercial FFC-A elements to:

Fresh JP-8 treated with only FFC-A and

Fresh JP-8 treated with FFC-A and trifluoroacetic acid.

The magnitude of the changes induced in the batch including trifluoroacetic acid were greater than those changes induced in the batch without. Both showed changes relative to untreated JP-8. These experiments were carried out using NMR. (See attachment)

Issues	•
100000	

1) Lack of aged fuel from SWRI is seriously impacting contract schedule and progress

Plans for next period:

- 1) Replant project schedule, milestone, and catalyst deliveries to SWRI to reflect delay in aged fuel delivery to APSI from SWRI. APSI needs firm schedule from SWRI.
- 2) Complete tests on samples delivered to Saybolt.
- 3) Perform PMR test series with available clean diesel fuel from SWRI to measure effect of exposure to FFC.
- 4) Continue development of high surface area catalysts.
- 5) Find suitable Lab to carry out ASTM tests for oxidation stability

William Lueckel

APSI Program Manager

6th MONTHLY PROGRESS REPORT

SWRI CONTRACT 3994750

MAY 6, 2003

Introduction:

This report covers work performed between the period of April 7, 2002 and May 5, 2003 by Advanced Systems International (APSI).

Accomplishments:

- 1) As a result of further investigation we were able to reach an understanding with Saybolt Laboratories to conduct the oxidation stability tests as well as the Karl Fisher (KF) on Diesel and JP-8 fuel. APSI is shipping the samples described in the March Progress Report to Saybolt. The understanding with Saybolt is contained in the attached memo from Dr. Berlin to Saybolt.
- 2) In addition to the samples prepared and reported previously we have also prepared samples of fresh Diesel fuel treated with FFC-A and fresh Diesel treated with FFC-A and "doped" with trifluoroacetic acid (0.01%) (to simulate oxidized fuel).

The fuel samples now awaiting test at Saybolt are:

- Fresh JP-8, untreated
- Fresh Diesel, untreated
- Fresh JP-8, treated with commercial Fitch Fuel Catalyst (FFC-A)
- Fresh JP-8, treated with FFC-A and "doped" with trifluoroacetic acid (0.01%) (to simulate oxidized fuel)
- Fresh Diesel, treated with commercial Fitch Fuel Catalyst (FFC-A)
- Fresh Diesel, treated with FFC-A and "doped" with trifluoroacetic acid (0.01%) (to simulate oxidized fuel)
- 3) In March we reported Dr. Berlin and the UCONN staff synthesized nanoparticles of very high surface area. This work continued in April. They were able to deposit the nanoparticles on silica and aluminum oxide in addition to copper

4) Dr. Berlin shipped a "Rocker Table" to UCONN (Attached memo). This will provide a more realistic means of simulating the movement of the catalyst in the fuel than the previous method of stirring the catalyst and fuel with a "magnetic stirring spoon"

Issues:

1) It will be helpful to the project to have oxidized diesel and/or jet fuel available as soon as possible

Plans for next period:

- 1) Perform tests on samples delivered to Saybolt.
- 2) Continued development of high surface area catalysts.
- <u>3)</u> Look at alternative approaches to obtaining oxidized fuel samples.

William Lueckel

APSI Program Manager

ADDENDUM TO THE MAY 2003 PROGRESS REPORT

Proton Magnetic Resonance Spectra (PMR) have clearly and unambiguously revealed that Fitch Fuel Catalysts (FFC) resuscitate both diesel fuel and JP-8 jet fuel. After 48 hour treatments of the fuels with FFCs of various formulations at ambient temperatures, we examine PMR spectra of the untreated fuels, of the fuels with 0.03% trifluoroacetic acid (v/v), of the fuels treated with FFC without acid, and the fuels treated with FFC and 0.03% acid (v/v).

Here is how we can deduce from the PMR spectra that the number of oxidized molecules in fuels is lessened after treatment with FFCs and with FFCs and acid, or to state this a different way, the number of saturated molecules (alkanes) is increased compared with unsaturated molecules (olefins, aromatics, etc.) after treatment.

There are too many molecules to resolve the molecular changes of each molecule. What we can resolve is the number of protons, i.e. the protons in each region compared with the number of protons in other regions. The numbers of each type of proton are represented, precisely, by the integrated area under each peak. For example, the PMR spectrum of Acetaldehyde, CH₃-CH=O shows two regions of peaks. One region forms a doublet, and the other region a quartet of peaks. The integrated area under the doublet is 3 times as great as the integrated area under the quartet, or 3:1. The area under the doublet is of course due to the CH₃ group, and the quartet area is due to the –CH=O proton. Now, we can identify the types of protons in the spectra, and measure the area under each group.

When we compare the aliphatic region of protons, to the protons in regions represented by olefinic and aromatic protons (the oxidized region of molecules), we find that in untreated fuels, the ratio of aliphatic to olefinic/aromatic is significantly lower than these ratios in fuels treated with FFCs and in fuels treated with FFCs and acid. It should be noted that the fuels treated with FFC and acid showed a much greater ratio of aliphatic to olefinic/aromatic than those fuels treated with FFC alone.

We are continuing to develop this analytical method, to aid us statistically and Quantitatively, to understand and improve our ability to resuscitate fuels. These recent observations allow us to make *deductions* that take us out of the realm of the hypothetical, to certainties in understanding what is really occurring.

AJB; 6-3-03

Volume 1 TAB 7.

7th MONTHLY PROGRESS REPORT

SWRI CONTRACT 3994750

June 6, 2003

Introduction:

This report covers work performed between the period of May 6, 2003 and June 6, 2003 by Advanced Systems International (APSI).

Accomplishments:

- 1) Testing commenced at Saybolt (the appropriate ASTM Oxidation Stability test and the Karl Fisher test). NMR testing of various catalyst fuel combinations continued at UCONN. The results of certain of the NMR tests have proven to be quite interesting a short write up of the significance of the tests prepared by D. Berlin is attached. The actual reports of the test are not included but are available if requested.
- 2) The following samples are being tested at Saybolt. In some instances Saybolt has reported their test results.
- 3) The fuel samples undergoing or completed at Saybolt are:
 - Fresh JP-8, untreated
 - Fresh Diesel, untreated
 - Fresh JP-8, treated with commercial Fitch Fuel Catalyst (FFC-A)
 - Fresh JP-8, treated with FFC-A and "doped" with trifluoroacetic acid (0.01%) (to simulate oxidized fuel)
 - Fresh Diesel, treated with commercial Fitch Fuel Catalyst (FFC-A)
 - Fresh Diesel, treated with FFC-A and "doped" with trifluoroacetic acid (0.01%) (simulates oxidized fuel)
- 4) A results matrix is being prepared to enhance comparisons of the results of the different tests. In addition several of the tests at Saybolt and UCONN will be repeated to determine the variance of the baseline tests (i.e. fuel with no catalyst treatment), and the effect of using the "Rocker Table" rather than the "magnetic stirring bar" as reported last month.
- 5) We are proactively seeking out other fuels for the test program while we await oxidized Diesel and JP-8 from SWRI.

Issues:

- 1) It will be helpful to the project to have oxidized diesel and/or jet fuel available as soon as possible
- 2) In the absence of the oxidized diesel and JP-8 fuel from we are searching for other sources of oxidized fuel and also for a source of bio-diesel, and marine diesel fuel.
- 3) APSI would benefit from some input and guidance from TACOM and / or SWRI as to the best direction to take in the absence of the oxidized fuel.

Plans for next period:

- 1) Continue tests of samples delivered to Saybolt, and NMR testing at UCONN.
- 2) Continued development of high surface area catalysts.
- 3) Look at alternative sources of fuels and alternative fuels for testing.

William Lueckel

APSI Program Manager

ADDENDUM TO THE JUNE 2003 PROGRESS REPORT

The UCONN staff turned out a large number of catalyst treatment runs on Jet fuel and Diesel fuel using the third catalyst system, Catalyst D of Catalysts A, C, and D. We analyzed the results using NMR, but did not find the same kind of trend we found in May on Catalyst C. The treated fuels were also sent off to Saybolt Labs for oxidation stability tests which have yet to be reported.

June's NMR results are still in the process of analysis, but the different trend will necessitate that in July we develop statistical analyses of the reproducibility of the NMR data, to determine confidence levels, precision and accuracy.

In June, we also started construction of a system that will allow us to evaluate the possible synergy between Fitch Fuel Catalysts and Magnetic Fields. This Catalyst system will be tested in July and will represent the fourth catalyst system tested.

In June, we also improved our catalyst tracking system by introducing a Code System for labeling catalysts and a matrix system for studying any trends that develop.

A. J. Berlin, 7-5-03

Volume 1 TAB 8.

8 th MONTHLY PROGRESS REPORT SWRI CONTRACT 3994750

July 8, 2003

Introduction:

This report covers work performed between the period of June 6, 2003 and July 3, 2003 by Advanced Systems International (APSI).

Accomplishments:

- 1) A large number of catalyst treatment "runs" on "fresh" JP-8 and Diesel fuel were conducted at UCONN using Catalyst D. (Catalyst A, C and D are included in the planned program).
- 2) Analysis of the NMR data for catalyst D show trends that differ from those we recorded in earlier tests with Catalyst C.
- 3) Construction of a laboratory level system to allow us to evaluate the possible synergy between Fitch Fuel Catalysts and magnetic fields was started at UCONN.
- 4) We modified our catalyst tracking system to include a Code System for labeling catalysts/fuel combinations (See Attachment A for detail). We are also preparing a test matrix to assist in organizing studies of data trends.

<u>Issues</u>:

- 1) It will be helpful to the project to have oxidized diesel and/or jet fuel available as soon as possible. Lack of aged (oxidized) test fuels from SWRI has prevented APSI from development and delivery of catalyst systems to SWRI as called for in Task 3 contract Schedule / Milestones
- 2) APSI would benefit from input and guidance from TACOM and / or SWRI as to the best direction to take in the absence of the oxidized fuel.

- 3) In the absence of the oxidized diesel and JP-8 fuel from we are searching for other sources of oxidized fuel and also for a source of bio-diesel, and marine diesel fuel.
- 4) Test results of treated fuels sent to Saybolt as per the June progress report, have not yet been reported. We expect to receive them in July

Plans for next period:

- 1) Continue tests of samples delivered to Saybolt, and NMR testing at UCONN.
- 2) Develop statistical analyses of the reproducibility of the NMR data to ensure reproducibility of the NMR data.
- 3) Continue search for alternative sources of "aged" and alternative fuels for testing.
- <u>4)</u> Initiate testing of laboratory system to study interaction of Fitch Fuel Catalysts in conjunction with magnetic fields on fuel.

William Lueckel

APSI Program Manager

ATTACHMENT A

CODE SYSTEM FOR LABELING FITCH FUEL CATALYST PROJECT SAMPLES

The labeling procedure uses letters in a certain order from left to right (Standard English).

1. The **First Letter(s)** identifies the fuel as follows:

D = diesel

G = gasoline

JA = Jet A

JE = Jet 8

M = Marine Diesel

If the fuel tested uses more than one letter, both letters are placed in parentheses, e.g., (JA).

2. The **Second Letter** identifies the FFC used.

A = Original formulation

B = Sn, Sb, Zn, Ag.

C = Sn, Sb, Bi, Pb.

D = Sn, Sb, Bi.

If no catalyst is used, as for a base line sample, empty parentheses are used in lieu of a letter, e.g., ().

3. The **Third Letter** describes whether an acidified or non-acidified fuel is treated.

a = acidified

n = non-acidified

4. The **Fourth Letter** describes the fuel as fresh or aged.

X = Aged or Modified

F = Fresh

5. The **Fifth Letter** describes some additional feature of the experiment.

M = Magnetic field

Other types of new features for future experiments will be described by other letters, so the Fifth Letter may change as the need arises.

The samples will be labeled with adequate labels, written or printed with indelible, non-fading ink.

Two examples of sample marking are now given: The first sample is Aged Diesel fuel, treated with catalyst C, non-acidified, with a magnetic field application:

DCnXM

The second example is Jet-8, not aged, acidified but not treated with any catalyst, but exposed to a magnetic field:

(JE)()aFM

AJB, 7-3-03

Volume 1 TAB 9.

9th MONTHLY PROGRESS REPORT SWRI CONTRACT 3994750

August 6, 2003

Introduction:

This report covers work performed between the period of July 6, 2003 and August 6, 2003 by Advanced Systems International (APSI).

Accomplishments:

- 1) We received notice from SWRI on August 4 that aged diesel, aged JP-8 fuel, and a one-quart sample of bio-diesel (B-100) was being shipped to APSI. The aged JP-8 is discolored and has a JFTOT breakpoint of 285 C by ASTM 3241. The aged diesel has a reflectance rating of 68 by ASTM D6468. The diesel fuel reflectance rating, prior to aging, was 85.
- 2) A number of N.M.R. analyses on diesel and JP-8 fuels were conducted as well as Gas Chromatographic (GLC) analyses on model compounds. All of the results were obtained after treating fuels or model compounds at ambient temperatures for 72 hours on a rocker table. Professor Suib (UCONN) and Dr. Berlin (APSI) have concluded the following from these tests:
 - a) In the presence of FF Catalysts some of the molecules in the whole fuel (JP-8 and diesel) undergo the process of chemical reduction while other molecules undergo the process of chemical oxidation.
 - b) N.M.R. is a useful technique to measure changes that occur due to the presence of various formulations of FFC in whole fuels.

- c) Gas Chromatography (GLC) is a useful technique for measuring changes that occur in model compounds of fuels due to the presence of various formulations of the FFC.
- d) Various chemical changes have been measured to occur in the presence of FFC. These include isomerization and cracking reactions.
- e) Acids may be co-catalysts and seem to enhance the activity of the FFC.
- 3) FFC-A has been incorporated into an MNO₂ "paper" in the form of nano-particles. The nano-particle MnO_2 paper has a surface area of $60m^2/g$. That is about 180,000 times greater than the present surface area of FFC-A.
- 4) Seven samples of fresh Diesel fuel and seven samples of fresh JP-8 fuel were tested by ASTM D-5304 for oxidation stability by measuring a precipitate, and by ASTM D-6304 (the Karl Fisher test for water content). Several blanks were included in the 14 samples. The test data has been received from Saybolt Labs and is now being analyzed. The data is available upon request.
- 5) We were able to obtain small quantities of aged gasoline and diesel fuel from automobile recycle facilities. We started testing the characteristics of these fuels and intend to utilize them.

Issues:

- 1. The arrival of the fuel from SWRI at APSI will facilitate completion of our test plan.
- 2. The ASTM D3241 tests performed on the aged JP-8 and the ASTM D6468 performed on the aged diesel supplied by SWRI differ from the ASTM test procedures performed on previously supplied JP-8 and diesel fuel. This will necessitate performance of equivalent tests on the previously supplied JP-8 and diesel in our possession.

Plans for next period:

- 1. Continue test and analysis of samples delivered to Saybolt, and NMR testing at UCONN.
- 2. Continued development of high surface area catalysts.

3.	Revise plan for the remaining work to reflect arrival of aged JP-8, Diesel, and bio-diesel fuels from SWRI.
W	illiam Lueckel
Aŀ	PSI Program Manager

Volume 1 TAB 10.

10th MONTHLY PROGRESS REPORT SWRI CONTRACT 3994750

September 6, 2003

Introduction:

This report covers work performed between the period of August 6, 2003 and September 6, 2003 by Advanced Systems International (APSI).

Accomplishments:

- 1. We set up an apparatus to simulate fuel pump systems in vehicles. This system uses a peristaltic pump that is easily flushed out when switching between fuels. It has valves to allow us to easily switch different catalysts in or out of the system; and one of two types of magnetic fields in or out of the system. It also allows injection of small amounts of acid co-catalysts into the circulating stream. Color changes are observed in gasoline under certain conditions. With the addition of the above apparatus, we are able to simulate both fuel pump fuel systems with in line FFC units (canisters) or FFC drop in units with our rocking table systems.
- 2. To improve the precision of our analysis of the N.M.R. data, Dr. M. Morton of UCONN has written a special computer program to standardize the way in which the integral areas are calculated. Previously the analysis was performed in different ways depending on the preference of the individual researcher.
- 3. An extremely interesting result has surfaced using nano-particles of one of the FFC formulations on a special molecular sieve support in gasoline. Relatively large changes in the N.M.R. are observed in gasoline treated with this unusual catalyst.

<u>Issues:</u>

We now have a surplus of data. Dr. Berlin will be spending much of his time analyzing, correlating, and interpreting the results.

Plans for next period:

- 1) Continue test and analysis of samples delivered to Saybolt, and NMR testing at UCONN.
- 2) Continued development of high surface area catalysts.
- 3) Revise plan for the remaining work to reflect arrival of aged JP-8, Diesel, and bio-diesel fuels from SWRI.
- 4) Continue to analyze model compounds by GC/MS, but we are now using some containing sulfur molecules (since at least some sulfur is present in all hydrocarbon fuels).
- 5) Continue our study of microorganism contaminated aged fuel.
- 6) Using new equipment available to UCONN we will be testing the acidity of fuel samples by ASTM D974-02.

William Lueckel

APSI Program Manager

ATTACHMENT

A large number of tests were made in September with a peristaltic pumping system that simulates a vehicle's fuel recirculation system. The setup permits variation in the type of fuel, the type of catalyst, and the introduction of acid into the fuel (to simulate the aging of fuel) or a magnetic field through the fuel.

Each test takes three days to complete. The fuel is pumped through the loop for 9 hours per day, passing over the catalyst, and through the magnetic field or not, as the case may be. The fuel remains in contact with catalyst throughout the 72 hours of the test. At the end of each day, the fuel is examined by N.M.R. The N.M.R. Spectra are compared with the spectrum of the untreated fuel.

In addition to the tests in the circulating fuel system, many fuel/catalyst combinations are tested in small "bottles" with and without acid. These tests are conducted without magnetic fields. Each sample is prepared and placed on a rocker for three days, and then NMR spectra data measured.

With all these variables, it is time consuming to deal with analyzing the data, but so far, we can see the following trends:

- 1). With moderately aged gasoline, Catalyst A is superior to Catalyst C in resuscitating the fuel.
- 2). With severely aged gasoline, Catalyst A transforms molecules of fuels to more branched structures.
- 3). Catalyst C may improve the cetane index number of fresh diesel after two days of pumping. This result will be confirmed by ASTM testing.
- 4). Catalyst C lowers the acid content of fuels much more rapidly than Catalyst A.
- 5). Catalyst A-1,(later designated as Catalyst X) the catalyst configuration that incorporates Catalyst A nano-particles onto an active substrate (see early section of this report), is active in gasoline in increasing its olefin content.

In October we will continue to use the pumping system to study FFC effects on aged diesel fuel and JP-8 produced by SwRI. In addition we will have these fuels, treated and untreated, tested with a full battery of ASTM tests by Saybolt Labs.

AJB, 10-07-03

Volume 1 TAB 11.

11th MONTHLY PROGRESS REPORT SWRI CONTRACT 3994750

October 6, 2003

Introduction:

This report covers work performed between the period of September 6, 2003 and October 6, 2003 by Advanced Power Systems International (APSI).

Accomplishments & Results:

- 46. We successfully manufactured a composite catalytic structure of very high surface area. The structure is composed of an inorganic molecular sieve with nano-particles of catalyst formula A embedded.
- 47. Tests of this composite catalytic structure (described in 1 above) in gasoline showed a much higher production of olefins than cast products comprised of either Catalyst A or Catalyst C.
- 48. Completed the construction and successfully tested the laboratory scale simulator of a recirculating fuel delivery system in an engine. A large amount of data was collected and analyzed. A number of trends were observed. These results are discussed further in the Attachment.
- 49. Confirmed, by NMR measurements, that the introduction of Catalyst A into aged gasoline results in changes to the molecular species in the gasoline.

<u>Issues:</u> The late delivery of aged diesel and JP-8 fuel has impacted our schedule to manufacture and deliver a large enough quantity of new catalyst to enable SWRI to conduct engine tests.

Plans for next period:

1) Manufacture a sufficient quantity of the composite high surface area catalyst to enable a thorough laboratory evaluation to be carried out.

2) Continue testing of Catalyst A and Catalyst C with aged diesel and JP-8 fuel. Some testing with fresh fuel will be conducted to confirm earlier results. Magnetic fields will be introduced as part of the evaluation of the catalysts, and possibly combinations of various ratios of catalysts A and C will be investigated.

William Lueckel APSI Program Manager

ADDENDUM TO THE 11TH MONTHLY PROGRESS REPORT OF SWRI CONTRACT 399475O, NOVEMBER 2003

October was an important month in terms of project progress and positive results.

First, we found that the molecular changes seen in NMR results for diesel fuels are mirrored by the Cetane Index of the samples as measured by ASTM D-4737. We find that when the ratio of the alkane proton area to the aromatic proton area, R_x , increases after treatment with FFC, this increase in R_x is mirrored by an increase in the Cetane Index. This permits us to rank various candidate catalyst formulations for diesel fuels using the very rapid Proton Magnetic Resonance (PMR) test rather than the longer, more expensive, ASTM test.

The test series also showed Catalyst C to be superior to Catalyst A in resuscitating the aged diesel fuel provided by SwRI to APSI.

Finally, the test results lead us to conclude the FFC promotes the dehydrogenation of non-beneficial impurity molecules in fuel while concurrently rehydrogenating oxidized fuel molecules.

We are continuing to study Jet Fuel and gasoline with various formulations of FFC to determine the most active ones for different fuels. In addition, we will try to use C^{13} NMR to elucidate other molecular transformations performed by various FFCs such as isomerizations.

AJB, Nov. 7, 2003

Volume 1 TAB 12.

12th MONTHLY PROGRESS REPORT SWRI CONTRACT 3994750 November 6, 2003

Introduction:

This report covers work performed between October 6, 2003 and November 6, 2003 by Advanced Power Systems International (APSI). This is the 12th and final monthly progress report required under the contract

Accomplishments & Results:

- We successfully manufactured a second composite catalytic structure of very high surface area. This structure is composed of the same inorganic molecular sieve with nano-particles of catalyst as reported last month. In this instance catalyst formula C was embedded.
- 2) NMR tests showed shifts in the composition of diesel fuel supplied by SWRI from exposure to catalysts A and C. The ratio of alkane / aliphatic proton area to the aromatic proton area increased after treatment with the FFC's in differing amounts depending upon the catalyst formula. This change allowed researchers to deduce from observing the NMR data that the Cetane index of the fuel increased as a result of exposure to catalyst A and C.
- 3) ASTM test D-4737 measuring the Cetane index of aged diesel fuel received from SWRI showed fuel treated with either catalyst A or catalyst C had a higher Cetane index than untreated fuel. Fuel treated with Catalyst C had a higher index than fuel treated with Catalyst A. This demonstrates FFC catalysts A and C are effective in resuscitating degraded Diesel Fuel.
- 4) These same ASTM tests corroborate the results of earlier NMR tests of the same fuel with the same catalyst family. The rank order of both NMR measures and ASTM measures were the same. That is, the treatment effectiveness improved from the blank, to A, to C. In addition to indicating C to be the superior catalyst

for diesel fuel, it also validates the NMR test as a surrogate for ASTM tests. The advantage of NMR is that it is much faster and much less expensive than ASTM testing and is therefore a good screening tool.

5) Continued the operation of the laboratory scale simulator of a recirculating fuel delivery system in an engine. A large amount of data was collected and analyzed. A number of trends were observed.

6) Further discussion of items 1 through 4 above is included in the Attached Addendum to the 12th Monthly Progress Report

<u>Issues:</u> The late date of arrival of aged diesel and JP-8 fuel has impacted our schedule to manufacture and deliver a large enough quantity of new catalyst.

Plans for next period:

1) Continue testing of Catalyst A and Catalyst C with aged diesel and JP-8 fuel. Some testing with fresh fuel will be conducted to confirm earlier results. Magnetic fields will be introduced as part of the evaluation of the catalysts, and possibly combinations of various ratios of catalysts A and C will be investigated.

 Complete project technical work. Prepare and deliver technical information SwRI may include in their final technical report to TACOM, as required by our subcontract. Initiate preparation of other closeout documents and planning documents as required by the contract.

William Lueckel
APSI Program Manager
Volume 1 TAB 13. –
Certification of APSI as to Progress Report 1-12 Completeness and Accuracy

The Subcontractor, ADVANCED POWER SYSTEMS INTERNATIONAL, INC. hereby certifies that, to the best of its knowledge and belief, the technical data delivered in the 12 monthly progress reports under Subcontract No. 399475O is complete, accurate and complies with all requirements of the Subcontract.

12/06/03

Name & Title of Certifying Official

Date

Inventions" have been reported.

I certify that the reporting party has procedures for prompt identification and timely disclosure of "Subject Inventions," that such procedures have been followed and that all "Subject

c. SIGNATURE

d. DATE SIGNED

2002/11/25

NAME OF AUTHORIZED CONTRACTOR/SUBCONTRACTOR

b. TITLE

President

OFFICIAL (Lest, First, Middle Initial) Best, Michael H.

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C ADDRESS (Include ZIP Code) Intl, (Pursuant to "Patent Rights" Contract Clause) (See Instructions on back) REPORT OF INVENTIONS AND SUBCONTRACTS SwRI 3994750 d. AWARD DATE | b. ADDRESS (Include ZIP Code) (DOWNYYYY) Southwest Research Inst. 6220 Culebra Rd. o. CONTRACT NUMBER d. AWARD DATE COMMALAL 4. REPORTING PERIOD (YYYYMMDD 3. TYPE OF REPORT (X one) a. INTERIM X b. FINAL Form Approved OMB No. 9000-0095 Expires Oct 31, 2004

Lakeville, Ct 06039

2002/11/25

SECTION I - SUBJECT INVENTIONS

San Antonio, Texas 78228-0510

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SUBCONTRACTOR'S RELEASE

Subcontract No. 3994750

Pursuant to the terms of Subcontract No. 399475O, and in consideration of the sum of Two hundred fifty thousand Dollars, (\$ 250,000) (total invoiced amount, including final invoice) which has been or is to be paid under the said contract to Advanced Power Systems International, Inc. (hereinafter called the Subcontractor) or its assignees, if any, the Subcontractor, upon payment of the said sum by Southwest Research Institute (hereinafter called the Prime), does remise, release, and discharge the Prime, its officers, agents and employees, of and from all liabilities, obligations, claims and demands whatsoever under or arising from the said Subcontract except:

- Specified claims in stated amounts or estimated amounts where the amounts are not susceptible of exact statement by the Subcontractor, as follows:
- 2) Claims, together with reasonable expenses incidental thereto, based upon the liability of the Subcontractor to third parties arising out of the performance of the said Subcontract, which are not known to the Subcontractor on the date of the execution of this release and of which the Subcontractor gives notice in writing to the Subcontract Administrator within the period specified in the said Subcontract.
- 3) Claims for reimbursement of costs (other than expenses of the Subcontractor by reason of his indemnification of the Prime against Patent Liability), including reasonable expenses incidental thereto, incurred by the Subcontractor under the provisions of the said Subcontract relating to Patents.

The Subcontractor agrees, in connection with patent matters and with claims which are not released as set forth above, that he will comply with all of the provisions of said contract, including without limitation those provisions relating to notification of the Subcontract Administrator and relating to the defense or prosecution of litigation.

IN WITNESS WHEREOF, this release has been executed this 25'th day of November, 2003.

Company Name Advanced Power Systems International, Inc.

By Title President	
Title Flesident	
I, Nora Dzenutis certify that I am the Secretary of the corporation named as Subcontractor in the foregoing release; that Michael H. Best who signed said release on behalf of the Subcontractor was the President of said corporation; that said release was duly signed for and in behalf of said scope of its corporate powers.	
Corporate Seal Nora Dzenutis	

Corporate Seal

SUBCONTRACTORS'S ASSIGNMENT OF REFUNDS, REBATES, CREDITS AND OTHER AMOUNTS

Subcontract No. SwrI 3994750
Pursuant to the terms of Subcontract No. 3994750, and in consideration of the reimbursement of costs and payment of fee, as provided in the said Subcontract and any assignment thereunder, the Subcontractor, Advanced Power Systems International, Inc. 558 Lime Rock Rd., Lakeville, CT 06039 (Subcontractor's name and address) (hereinafter called the Subcontractor) does hereby:
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Assign, transfer, set over and release to Southwest Research Institute (hereinafter called the Prime), all rights, title and interest to all refunds, rebates, credits and other amounts (including any interest thereon), arising out of the performance of the said subcontract, together with all rights of action accrued or which may hereafter accrue thereunder.
Agree to take whatever action may be necessary to effect prompt collection of all refunds, rebates, credits and other amounts (including interest thereon) due or which may come due, and to promptly forward to the Subcontract Administrator checks (made payable to Southwest Research Institute) for any proceeds collected. The reasonable costs of any such action to effect collection shall constitute allowable costs when approved by the Subcontract Administrator as stated in the said contract and may be applied to reduce any amounts otherwise payable to the Prime under the terms hereof.
Agree to cooperate fully with the Prime as to any claim or suit in connection with refunds, rebates, credits or other amounts due (including any interest thereon) to execute any protest, pleading application, power of attorney or other papers in connection therewith; and to permit the Prime to represent him at any hearing, trial or other proceeding, arising of such claim or suit.
IN WITNESS WHEREOF, this assignment has been executed this25'thday of
November , 2003 .
Company NameAdvanced Power Systems International, Inc.
By Michael H. Best
Title President
I, <u>Nora Dzenutis</u> , certify that I am <u>Secretary</u> of the corporation named as Subcontractor in the foregoing release; that <u>Michael H. Best</u> who signed the release on behalf of the Subcontractor was the <u>President</u> of said corporation; that said release was duly signed for and in behalf of said corporation by authority of its governing body and is within the scope of its corporate powers.

ADVANCED POWER SYSTEMS INTERNATIONAL INC.

558 Lime Rock Road Lakeville, CT 06039 tel 860-435-2525 fax 860-435-2424 Website – www.fitchfuelcatalyst.com

March 2004

RE: SwRI Contract number 399475O – Advanced Power Systems International, Inc.

APSI's final submittal to SwRI for this contract consists of two volumes.

Volume II

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- 23. Final Report of experiments conducted, observations made and conclusions drawn during the course of the contract and work.
- 24. Figures 1-11
- 25. Appendices 1-12
- 26. Certification of APSI as to Final Report Completeness and Accuracy
- 27. Plans for catalyst scale-up, laboratory testing, and field tests

This document contains INDUSTRIAL PROPRIETARY Information that is the property of Advanced Power Systems International, Inc. Lakeville, Ct. (APSI) and shall not be disclosed to anyone other than the intended recipient and shall not be duplicated, used, or disclosed in whole or in part for any purpose other than to inform the intended recipient. The INDUSTRIAL PROPRIETARY Information contained in the document is subject to INDUSTRIAL PROPRIETARY Agreements between APSI and Southwest Research Institute (SwRI) dated December 17, 1998 and January 9, 2004. In accordance with 18 USC 1905 Employees of the US Government and the Government shall have the right to duplicate, use or disclose this data to the extent provided in any contract between Advanced Power Systems International, Inc. and the US Government. This restriction does not limit the Government right to use the information contained in the data if it is obtainable from another source without restriction. The information and data subject to this restriction are blacked out and marked INDUSTRIAL PROPRIETARY and appear on pages A39 - A40 and A44.

REPORT OF FUEL CATALYST RESEARCH AND EVALUATION

Ву

ADVANCED POWER SYSTEMS INTERNATIONAL, INC., NOV. 2002 - NOV. 2003

For

SOUTHWEST RESEARCH INSTITUTE - Contract Number 3994750

Signature page

Alvin Berin PhD Advanced Power Systems International, Inc.

Date 3/15/04

Board of Trustees Distinguished

Professor

Department of Chemistry University of Connecticut Storrs, CT 06269-3060

Tab 18 REPORT OF FUEL CATALYST RESEARCH AND EVALUATION

By

ADVANCED POWER SYSTEMS INTERNATIONAL, INC., NOV. 2002 - NOV. 2003

For

SOUTHWEST RESEARCH INSTITUTE - Contract Number 3994750

EXECUTIVE SUMMARY

Program Goal

The goal of this program was to "extend the capability of the patented precombustion Fitch Fuel Catalyst (FFC) to diesel and JP-8 fuel with special emphasis on retarding, reversing, and or preventing oxidation and microbiological growth in these fuels."

Accomplishment(s):

The research effort was successful in the following areas:

I. Laboratory experiments

- 1. Developed laboratory methods that could rapidly demonstrate the effectiveness of various FFC formulations.
- 2. Demonstrated that FFC catalyst activity reversed auto oxidation of diesel and JP fuel.
- 3. Demonstrated that FFC catalysts are capable of altering structure of molecules in fuels that were the subject of the program.

II. Next Generation Prototype Development

- 4. Produced (nano-scale) versions of the FFC formulae A and C.
- 5. Selected a candidate substrate for high surface area (nano scale) version of fuel catalyst for future development.

III. Plan for future work

6. Prepared a plan for laboratory work of next generation high surface area and enhanced activity catalysts and associated combustion testing.

INTRODUCTION

Advanced Power Systems (APSI) was enjoined to produce a minimum of three fuel catalysts (FFCs) that would improve the performance of diesel fuels and jet fuels.

In order to assist in this we hoped to develop laboratory methods that could rapidly demonstrate the effectiveness of various FFC formulations; to show that FFCs could resuscitate aged fuels and auto-oxidized fuels; and demonstrate that FFCs will inhibit the growth of microorganisms that metabolize fuels.

Our studies were carried out in the Chemistry Department of the University of Connecticut (UCONN) at Storrs, CT. We used this fine Department for several reasons. The Department is headed by Distinguished Chairman, Professor Steven Suib, who is familiar with our fuel catalysts from prior research with APSI and is extremely qualified in this area. This Chemistry Department is equipped with fine, modern analytical equipment; and we have access to 4 graduate students interested in this research that could assist us in these endeavors.

Our goal was to develop laboratory methods that could rapidly allow us to judge the ability of FFCs to:

- 1. Modify jet fuels and diesel fuels in such a way that they will burn more efficiently and
- 2. Resuscitate fuels that had become oxidized due to auto-oxidation, exposure to ambient ozone, and bacteriological processes.

These problems occur in fuels as a result of storage and shipment. We also were aware that if we could demonstrate resuscitation, then our FFCs could also function as prophylactics to increase the storage life of diesel and jet fuels indefinitely.

In order to confirm the results of our laboratory procedures, whenever possible, we compared the lab results with ASTM tests. We will discuss the meaning of these correspondences at length in some of the following sections.

THE CATALYSTS

For these studies we developed Fitch Fuel Catalysts labeled **A**, **B**, **C**, **D**, and **X**. **B** was eliminated early in this study because of its morphology, and **D** was eliminated because of its similarity to **C**. FFC-**X** differs in its physical construction from the other catalysts, because it is composed of a high surface area (over a thousand times greater) nano scale formulation of FFC-**A** or FFC-**C** deposited on a support structure. We observed that the support entered into the chemical activity. FFC-**A**, FFC-**C** and FFC-**X** will be discussed in the section titled Results.

In addition, we developed a pumping system that simulates fuel re-circulation systems in vehicles. We studied the influence of magnetic fields from permanent magnets in this re-circulation system in conjunction with FFC-A and FFC-C. These combined systems (FFC-A+ Mag) and (FFC – C + Mag) are considered separate catalyst systems. **METHODOLOGY**

Fuels such as gasoline, diesel fuel, and jet fuel are composed of several hundred different molecules. Most of these molecules are hydrocarbons. Many of the hydrocarbons are different when different crude oils, the source of these fuels, are compared. The exact make up of the fuels depends on a myriad of factors. Some of the factors that determine differences are the source of the crude oil, refinery practice, processing parameters, and the time, temperature, and humidity of storage.

Because of this complexity, analytical methodologies that would allow us to map the exact molecular changes after treatment of fuels with FFCs do not exist. Even the best Gas Chromatograph / Mass Spectrometer (GC/MS) cannot resolve many of the individual molecules, because the hydrocarbons are too similar.

We can, however, examine the general nature of the fuel in a semi-quantitative way by means of Nuclear Magnetic Resonance (N.M.R.) Spectroscopy. More specifically, in our case we can examine each fuel, before and after treatment with FFC's, by Proton Magnetic Resonance (P.M.R.) Spectroscopy. We can actually count the number of hydrogen atoms of various types by integrating the area under the peaks of those types to find the ratios of the numbers of hydrogen atoms of each type. The P.M.R. fuel samples, both before and after catalyst treatment were run in solutions of deuterated chloroform, CDCl₃.

In addition to the P.M.R. studies we chose certain pure compounds we called "Model" compounds to represent gasoline, diesel fuel or jet fuel, or some impurity molecule. After catalyst treatment, we examined the "Model" Compound using P.M.R. and GC/MS.

We developed two ways of <u>exposing</u> fuels to FFCs. One way was to use a rocker table on which brown bottles of fuel containing catalyst were tipped one way or another, at ambient (room) temperature, for three days. This motion simulated FFC "drop in units" that are in actual road use. The second method utilized a pumped system built on a chemical rack inside of a chemical hood. A variable speed peristaltic pump was used to pump the fuel through special canisters that contained the catalysts. Fuel was pumped from the reservoir through the system and back to the reservoir, thus simulating vehicle fuel pumps. Fuel was in contact with the catalyst for 78 hours, but pumping was carried out only for 21 hours of those 78 hours, simulating intermittent vehicle use. Valving could direct the flow through special paths, some containing magnetic fields in certain configurations. This second method was also operated at ambient temperatures, and samples were removed every 24 hours for testing.

At the start of our program, we exposed fuels to FFCs at about 65°C in an attempt to accelerate the rate of reactions in order to accomplish as many experiments as possible during the duration of the program. This is within the range (65°C - 100°C) of temperatures of "heavier" fuel oils used in generators or boilers prior to combustion in order to insure good handling in fuel systems. FFC activity was measured at that temperature, but we felt it doubtful that such tests would represent actual field usage of the diesel and JP fuels that were the subject of this investigation, so no results from those studies will be presented.

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Many of the fuels were also tested by the "Liqui-Cult" procedure for bacterial and fungal contamination. These tests were performed at UCONN also. All of the samples, including the artificially aged diesel and jet fuels supplied to APSI by SWRI, had unimportant amounts of microorganisms, which did not interfere with the N.M.R. work, or the measurement of catalyst activity. Previous work at UCONN, where microorganisms were cultured on fuels, and the colonies counted, showed that FFCs brought the growth of microorganisms to a dead stop. Therefore no further mention of microorganisms will be presented in the Results Section.

Outside Laboratory Tests

The Saybolt Laboratory of New Haven, CT (an outside laboratory) was used to perform:

Karl Fisher (KF) method for water in fuels, and

Cetane Index in diesel fuels ASTM D-4737

Oxidation Stability Tests on diesel fuels
JFTOT Tests on jet fuels.

ASTM D-5304
ASTM D-3241

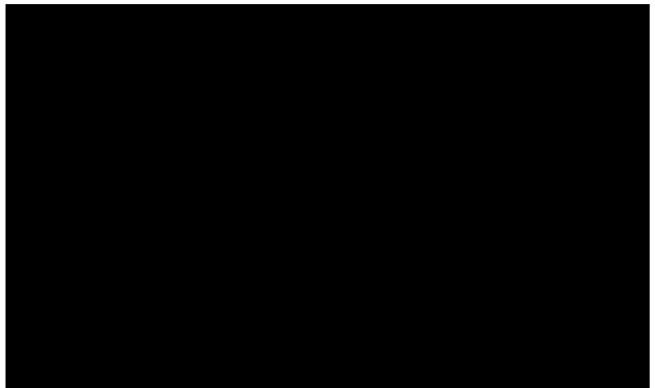
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DIFFERENCES IN COMPOSITION OF IDEAL AND AUTO OXIDIZED FUEL

Diesel and jet fuels are popular because of their properties in combustion. The best fuels for diesel and jet engines are composed of long chain aliphatic hydrocarbon molecules also called alkanes. These are described in "Fuels and Lubricants Handbook", George E. Totten, Ed., ASTM, 2003.

Combustion is almost complete oxidation. The end products of complete oxidation are mainly carbon dioxide (CO_2 gas) and water (H_2O). Auto-oxidation is partial, or slow, combustion. The end products of auto oxidation or partial combustion are olefins, alcohols, ketones, and organic acids. When auto oxidation occurs fuel becomes less effective. Less energy, less mileage, poorer combustion, more gums, more soot, more pollution, and more maintenance result as fuel ages.





In order to test our FFCs on aged fuels, we required a suitable quantity of aged diesel and JP-8 fuels, which SWRI offered to produce. These fuels arrived at APSI in month number 8 of a 12-month program, so the aged diesel and jet fuels entered our testing process late in the research. Testing on these fuels yielded important results. We could have done many more studies had these fuels been in our possession for the duration of the program. These aged diesel and jet fuels produced by SWRI did not have any more bacteria and fungi than the standard non-aged fuels and the Cetane Index of the aged diesel fuel at 48.8 showed it to be of standard quality (see Appendix #1 and compare it to Appendix #4, an off road diesel sample blank with a Cetane Index of 40.6.

RESULTS

The text in this Results section refer to PMR Spectra, ASTM Tests, and color change.

The P.M.R. spectra on treated and untreated fuels are in the Figures section.

The examples of color change are presented in the Figures section.

The ASTM tests on treated and untreated diesel fuels for Cetane Index, Oxidation Stability, and results of the ASTM JFTOT performed on JP fuels, are presented in the <u>Appendices</u>.

All fuel samples prepared for PMR or ASTM or color change analysis that are presented in the Figures and Appendices of this report were prepared using the rocker table agitation method (described on page 5) except for those fuel samples used in Appendix 6 and 7 which were prepared in the re-circulation system where we exposed diesel fuel to a catalyst and a magnetic field.

In general, we found that our hypothesis is correct. In every fuel studied, JP-8, diesel fuel, and gasoline, the alkane region in the P.M.R. increases with respect to the aromatic and olefinic region, thus increasing the $\bf R$ value. We examined hundreds of spectra, and ran several statistical studies to find that with catalysts FFC-A, FFC-C, and FFC-A + Magnetic Field, the effect is there. With some fuels, the effect is quite small. The reason is as follows: When we take pure n-dodecane, a 12 carbon hydrocarbon, $C_{12}H_{26}$, and treat it with one of our FFCs, we see no change in the P.M.R. We have repeated this with several pure model compounds. The reason is that in order for FFCs to operate, they require labile hydrogen atoms. For the same reason, FFCs protect the fuel against oxidative degradation because oxygen and ozone attack hydrocarbons to form products that have labile hydrogen atoms. These allow the FFC to reverse the process and to also react with dissolved oxygen and ozone perhaps producing water. Thus, we can increase the "shelf life" of fuels greatly without using additives.

All of the data produced during the years work is too voluminous to present in this report. The material we will present to the readers of this report is chosen to allow the reader to understand how we came to our conclusions by examining some of the spectra and by comparing conclusions drawn from the spectra to ASTM tests of various types that offer similar conclusions. In addition, with a given fuel, if one of our particular catalysts, say FFC-A as an example, gives a greater **R** value than another catalyst with that same fuel, say FFC-C, we must conclude that catalyst A is more effective for that particular fuel. We do not fully understand all the reasons why one catalyst may be better than another on a fuel but we will report results based on the measured **R** values.

P.M.R. Spectra: JP Fuel

A typical P.M.R. spectrum of aged JP fuel is shown in Figure #1. This is a blank run #1. 3 analyses were performed for statistical purposes. Examining Figure #1, we can see that the ratio, R=23.38. Were we to calculate R for the three spectra, we find that $R_{xbar}=23.32$ (Mean Value).

Figure #2 is a P.M.R. spectrum of the same aged JP fuel after treatment with FFC-A for three days on a rocker table. In Figure #2, R=28.64. Four runs of this sample were run for statistical purposes, where the mean value, R_{xbar} =26.83. The difference between exposed and unexposed samples, 3.5, tells us that about 80% of the hydrogen atoms have been transferred from the unsaturated regions to the aliphatic regions.

	Aged JP Fuel (Blank vs with FFC A)	R	R_{xbar}
Figure 1	Aged JP Fuel - Blank	23.38	23.32
Figure 2	Aged JP Fuel + FFC A	28.64	26.83
	Change	5.34	3.51

To demonstrate the differences between catalyst FFC-A and FFC-C in their effects on aged jet fuel, we include Figure #5, (for comparison with Figure # 2), the first of three spectra of non-acidified aged jet fuel after treatment with FFC-C, where R=24.25, $R_{xbar}=25.01$.

	Aged JP Fuel (Blank vs + FFC A vs + FFC C)	R	R_{xbar}
Figure 1	Aged JP Fuel – Blank	23.38	23.32
Figure 2	Aged JP Fuel + FFC A	28.64	26.83
Figure 5	Aged JP Fuel + FFC C	24.25	25.01

We have also added acid to this aged JP fuel to simulate the aging process even more. The blank sample in this case is aged JP fuel with 0.02% trifluoroacetic acid. Figure #3 represents the blank P.M.R. In this case R=25.25, and in the mean value of three runs, $R_{xbar}=24.88$. In Figure #4, after treatment of the acidified jet fuel with FFC-A, R=28.08, and for three runs, $R_{xbar}=27.24$. About 63% of the hydrogen atoms have been transferred.

	Aged JP Fuel + Acid (Blank vs FFC A)	R	R _{xbar}
Figure 3	Aged JP Fuel + Acid - Blank	25.25	24.88
Figure 4	Aged JP Fuel + Acid + FFC A	28.08	27.24
	Change	2.83	2.36

Independent outside laboratory results of FFC exposed and non-exposed JP fuel, using ASTM test JFTOT (ASTM D-3241) shows that the test results are virtually identical. Both the FFC-C exposed aged JP fuel and the unexposed aged JP fuel have tube deposit ratings of <1, while the exposed fuel has a pressure drop of 1 mm. The JFTOT tests on the blank aged JP-8 and the FFC-C treated JP-8 are shown in Appendix 9 and Appendix 10, respectively.

(It should be noted that we have determined that the blank aged JP-8 sample shown in Appendix 9 had been mislabeled. The ID given here is correct. It is an untreated aged JP-8. This is the only sample that has been mislabeled upon return from Saybolt Labs.

Aged Diesel Fuel and Commercial Off Road Diesel Fuel

P.M.R. Spectra, ASTM D-4737 Cetane Index, ASTM D-5304 Oxidation Stability

We have demonstrated the FFCs effect on aged diesel fuel in a similar manner as we did in the section on jet fuel, by examining the PMR Spectra. The **R** values are calculated in the same manner as before by dividing the integral of the aliphatic region by the integral in the aromatic region. The increases in the **R** value after treatment are clearly evident, and the **R** Values correspond to the Cetane Index values measured by ASTM D-4737. The corresponding values allow PMR to be used as a quick way to determine the Cetane Index number. The method employed was to:

- 1. Run the P.M.R. spectrum of the Diesel Fuel dissolved in deuterated chloroform (CDCl₃) and calculate the **R** Value.
- 2. Then calculate the Cetane Index value by using the following equation:

CETANE INDEX =
$$(1.76 \times R) + 19.8$$

The ASTM cetane index test D-4737 attempts to measure the real content of the cetane in diesel fuel by measuring the density and the distillation points of an *unadulterated fuel*. Additives can effect the measurement of cetane by manipulating the density and boiling point numbers, in effect, influencing the cetane values artificially. Since the FFC is not in any way an additive, that is it does not dissolve in the sample, it cannot "fool" the ASTM index test.

Diesel fuel aged by SWRI (the blank) had a Cetane Index of 48.8. The cetane index for commercially available Off-Road Diesel Fuel we procured in this program was 40.6. Both of these fuels have had the cetane values improved, as measured by both the ASTM D-4737 and the **R** value calculation, after treatment with FFCs. Figure 6 and Figure 7, a blank off road diesel fuel and a fuel exposed to FFC-C, respectively, show **R** values of 11.7 and after treatment 11.8. The corresponding ASTM D-4737 Cetane Index tests are Appendix 4 and 5 respectively.

Figure	Commercial Off Road Diesel Fuel	R	Appendix	Cetane
6	Off Road Diesel Fuel – Blank	11.7	4	40.6
7	Off Road Diesel Fuel + FFC-C + Mag	11.8	5	40.8

Figure	Aged Diesel Fuel Supplied by SwRI	R	Appendix	Cetane
	Aged Diesel Fuel – Blank		1	48.8
	Aged Diesel Fuel + FFC-A		2	49.0
	Aged Diesel Fuel + FFC C		3	49.2
8	Aged Diesel Fuel + Acid	16.03	6	48.3
	Aged Diesel + Acid + FFC-A + Mag	17.1	7	48.5
9	Aged Diesel Fuel + Acid + FFC C	16.83	8	48.6

Acidified diesel fuel exposed to catalyst FFC- A + Magnetic Field (Appendix 7) experienced an increase in both the **R** value and Cetane Index in comparison to the untreated sample (Figure 8 - Appendix 6)

Diesel Fuel Oxidation Stability ASTM D-5304

We performed ASTM D-5304 Oxidation Stability tests but we are not presenting a section on this data. From our perspective this test procedure is not informative regarding the nature of the catalytic action. This test did not illuminate changes in molecular structure and actual chemistry the way that the P.M.R. tests did. The gasoline oxidation test ASTM D-525 correlates with our P.M.R. results very well. It is a better indicator of actual chemical change.

Appendices 11 and 12 Oxidation Stability Tests of Fresh Diesel before and after exposure to FFC – C are included. Oxidation Stability was improved post exposure to the catalyst, which is consistent with resuscitation.

Acid Tests: ASTM D974-02

The <u>Tables 1,2, and 3</u> list results of acid tests, (ASTM D- 974-02) on various treated and untreated samples of diesel and JP fuels.

INDUSTRIAL PROPRIETARY



REPORT ON THE ACID NUMBER TESTS ON TREATED AND UNTREATED FUELS FOR APSI (Part II)

By Ruma Ghosh

25th September 2003

Acidity of the fuels was tested by the standard **ASTM D 974-02** method. Both catalytically treated and untreated fuels were subjected to the acidity tests. All samples of the Jet A fuel and aged gasoline # 5 were tested for acidity, which has been represented by the acid number. Each sample was tested for acid number five times and the statistical average, with standard deviation and the range are reported here.

Results:

Description of sample	Mean acid number of five runs (mg KOH/ g sample)	Range of acid no.	Standard deviation
Jet Fuel A	0.0478	0.0447 - 0.0555	0.0039
Jet Fuel A + catalyst A and no acid	0.0469	0.0457 - 0.0474	0.0006
Jet Fuel A + no catalyst A + acid	0.5876	0.5717 - 0.6010	0.0120
Jet Fuel A + catalyst A + acid	0.1998	0.1905 - 0.2088	0.0058
Jet Fuel A + catalyst C and no acid	0.0478	0.0471 - 0.0487	0.0007
Jet Fuel A + catalyst C + acid	0.1688	0.1640 - 0.179	0.0052
Aged gasoline #5	0.2280	0.2190 - 0.2387	0.0076
Aged gasoline # 5 + catalyst A and no acid	0.2465	0.2352 - 0.2596	0.0110

Table 1

REPORT ON THE ACID NUMBER TESTS ON TREATED AND UNTREATED FUELS FOR APSI

By Ruma Ghosh

11th September 2003

Acidity of the fuels was tested by the standard **ASTM D 974-02** method. Both catalytically treated and untreated fuels were subjected to the acidity tests. All samples of the **aged diesel** (15 months old) were tested for acidity, which has been represented by the acid number. Each sample was tested for acid number five times and the statistical average along with standard deviation are reported here.

Results:

Description of sample	Mean acid number of five runs (mg KOH/ g sample)	Standard deviation
Aged Diesel (15 months old)	0.107	0.006
Aged Diesel (15 months old) + catalyst A and no acid	0.104	0/010
Aged Diesel (15 months old) + no catalyst A + acid	0.489	0.010
Aged Diesel (15 months old) + catalyst A + acid	0.466	0.046
Aged Diesel (15 months old) + catalyst C and no acid	0.105	0.014
Aged Diesel (15 months old) + catalyst C + acid	0.378	0.005

Notes:

Alcoholic potassium hydroxide was standardized with standard potassium acid phthalate each day before acidity testing.

Acidity testing with treated and untreated jet fuels is presently going on.

Table 2

REPORT ON THE ACID NUMBER TESTS ON TREATED AND UNTREATED FUELS FOR APSI (Part ${ m IV}$)

By Ruma Ghosh

30th October 2003

Acidity of the fuels was tested by the standard **ASTM D 974-02** method. All samples of the DIESEL were treated for acidity, which has been represented by the acid number. Each sample was tested for acid Number five times and the statistical average, with standard deviation and the range are reported here.

Results:

Description of sample	Mean acid number of five runs (mg KOH/ g sample)	Range of acid no.	Standard deviation
Diesel	0.0264	0.0212-0.0230	0.005
Diesel + catalyst D and no acid	0.0215	0.0211-0.0217	0.0002
Diesel + no catalyst D + acid	0.5054	0.4963-0.5139	0.0053
Diesel + catalyst D + acid	0.2148	0.1980-0.2276	0.009
Diesel + catalyst C and no acid	0.0214	0.0212-0.0216	0.0001
Diesel + catalyst C + acid	0.1942	0.1821-0.2071	0.0090
Diesel from Southwest refinery	0.0490	0.0431-0.5700	0.0064

Notes:

Alcoholic potassium hydroxide was standardized with standard potassium acid phthalate each day before acidity testing.

Table 3

Other Tests and Observations

Color changes in hydrocarbons are often indicative of molecular changes. Figure 10 shows the change in color of a gasoline sample before and after exposure to FFC-A and magnets in circulating pumped system.

Figure 11.

Simulating the aging process in a pure, pale amber colored jet fuel by adding a small amount of trifluoroacetic acid, turns the fuel a dark brown as seen in figure 11b. Adding FFC-A to the flask reverses this color change by transforming the jet fuel over a several minute period to a clear, colorless jet fuel as shown in Figures 11a.

Several model compounds were analyzed by PMR and GC/MS before and after exposure to FFCs. Most of the compounds remained unchanged, but there appeared to be some tailing in acetophenone.

Treatment of Gasoline with FFC

This section presents supplemental information not funded by contract #399475O. It is in this separate section because jet and diesel fuels were the focus of contract #399475O. The methods developed for that study, however, grew out of our experience in studying the effects of FFCs on gasoline. The P.M.R. method, in which the **R** value is measured, tells us whether the aged, partially oxidized gasoline can be effectively resuscitated.

The experiments carried out with gasoline reveal that **R** values increase when the gasoline is treated with FFC-A, FFC-B, FFC-D, and FFC-A with magnets. However, treatment with FFC-X reduced the **R** value. In other words, the aromatic and olefinic content of the gasoline increases, with respect to the aliphatic region. For some gasolines, this reversal of reduction, or oxidation, can be important to increase the octane rating of the fuel *in the vehicle*, by increasing the aromatic content (e.g. benzenes and toluenes) in situ. FFC-X is worthy of further investigation in this area.

CONCLUSIONS

The experiments performed are entirely consistent with dehydrogenation/rehydrogenation reactions of oxidized fuels. The following statements can be made with a high degree of confidence:

- 1. A P.M.R. method, using a ratio, R, has been developed that proves that FFCs transform diesel and Jet fuels.
- 2. FFCs resuscitate diesel fuel and jet fuel that is aged and partially oxidized. We reach this conclusion due to parallel results of 3 different experiments, the Ratio, **R**, the ASTM cetane index, and change in fuel color. (See 5, 6, and 11.)
- 3. Cetane Index increases observed after FFCs treat diesel fuel corroborates the P.M.R. method. The ASTM Cetane Index increases measured in diesel fuel were small, but the fact that we are find two methods that give parallel results and correlate, increases certainty the conclusions are correct. The same result was observed in over one hundred P.M.R. experiments, adding to our assurance this is **not a random** phenomenon.
- **4.** P.M.R. is a suitable method for rapidly testing the efficacy of new catalysts.
- 5. The presence of FFCs in a fresh diesel or jet fuel vessel or tank have the potential to extend its shelf life and keep it fresh, indefinitely.
- **6.** FFC-C is superior to FFC-A with diesel, but FFC-A plus magnet is superior to FFC-C with jet fuel. Durability tests of both should be performed.
- 7. FFC-X is not suitable for diesel or jet fuel, since it has some oxidizing character. It may be suitable for producing high-octane gasoline in the tank. Its behavior differs from our experience with FFC-A and gasoline. We are inclined to attribute this difference to the substrate employed in X.
- **8.** ASTM D-974-02, Acidity Test is suitable for quality control for production purposes at the plant level for large-scale production of FFC's.
- 9. JFTOT tests of aged jet fuel show very little difference between exposed and unexposed fuel. This is not surprising because the JFTOT test has no molecular correspondence with the P.M.R. results. The P.M.R tests on jet fuel do show considerable improvement, however, after treatment with FFCs.

ADDITIONAL INSIGHTS

- 1. Initial Carbon Thirteen (C^{13}) N.M.R. shows possible isomerization of fuels.
- 2. Gas Chromatography studies of bubbling phenomena when FFC contacts fuels indicates that some cracking of gasoline occurs to off gas small carbon molecules such as methane, ethylene, and ethane.
- 3. Isomerization mechanisms may be operating in gasoline to produce more branched molecules. Migration of methyl groups to form additional toluene is observed. Disproportionation of some molecules may be occurring, where one part of a molecule is oxidized and another part is reduced. This was discussed among the researchers at length, in terms of hydride shift mechanisms.
- **4.** FFC seems to react much differently with the highly branched, olefinic and aromatic molecules in gasoline than the predominately straight chain molecules in diesel fuel and jet fuel.

All of the above possibilities warrant further investigation and are included in our planned research studies. One study that has not been described is the important use of FFC-A in suppressing the growth of microorganisms in diesel and jet fuel. We have no data, yet, regarding the properties of other iterations of FFCs, such as FFC-B, FFC-C, FFC-D, and FFC-X in controlling the growth of microorganisms. The Liqui-Cult system, of semi-quantitative measurement bacteria and fungi in fuels is one we have used before, and one that is rapid and effective.

CANDIDATE FFC's RECOMMENDED FOR COMBUSTION TESTING

As the bulk of evaluations in this test program were performed on FFC-A and FFC-C in combination with magnetic fields and they were demonstrated to be effective in certain circumstances we recommend and will supply those to conclude this contract.

CANDIDATE FFC's RECOMMENDED FOR FURTHER TESTING INCLUDING COMBUSION

Based upon these observations at the chemical level we suggest that the following list of catalysts be produced for further study of their influence on fuels and changes they can bring to bear on the combustion process.

- 1. For JP fuels FFC formula: FFC-B and FFC-D, FFC-A-on a Cordierite substrate
- 2. For Diesel Fuels FFC formula: FFC-C incorporating a Solid Acid
- 3. For Gasoline Fuels FFC formula: FFC-XM, FFC- A- incorporating a Solid Acid

See Separate File Tab 19 Figures

See Separate File Tab 20 Appendices

Vol 2 Tab 21 Certification of APSI as to Final Report Completeness and Accuracy

The Subcontractor, ADVANCED POWER SYSTEMS INTERNATIONAL, INC. hereby certifies that, to the best of its knowledge and belief, the technical data delivered herewith under Subcontract No. 399475O is complete, accurate and complies with all requirements of the Subcontract.

3.31.2004 William Suichel PROJECT MANAGE

te Name & Title of Certifying Official

Volume 2 Tab 22

PRELIMINARY PLAN FOR CATALYST SCALE-UP, LABORATORY TESTS, AND FIELD TESTS

Plan Overview

This plan builds upon and augments the work APSI is engaged in for Southwest Research Institute contract number 399475O.

The existing APSI contract program is composed of three tasks:

TASK 1. Catalyst Research

TASK 2. Laboratory Tests

TASK 3. Planning, Reporting, and Administration

The plan expands the existing program by including additional fuel / catalyst analytical research and evaluations on a broad array of fuels and combustion devices.

The plan envisions a cost-shared program of 4 Tasks conducted in 2 Phases.

Phase 1

- 1. Catalyst research and formulation
- 2. Catalyst scale-up and activity verification / Combustion Testing

Phase 2

- 3. Combustion Unit and Engine Testing / Optimization
- 4. Plans, Reports and Administration

Activities in tasks 1-3 are iterative. The output from engine and combustion device testing provides data and input to fuel catalyst improvement through modification of fuel catalyst composition, surface area, and through incorporation of other elements (e.g. magnets).

Tasks 1 and 2 of Phase 1 would be performed primarily at the University of Connecticut (UCONN) under the direction of Dr. A. Berlin of APSI and Dr. Steven Suib of UCONN.

Task 3 in Phase 2 (combustion testing) would take place at laboratories or test facilities most suitable for each investigation. Based upon informal communications received to date APSI would plan to select from the following list depending upon the specialty(s) of the particular lab for each task:

- TIAX (formerly Arthur D. Little) Cambridge Mass
- Southwest Research Institute, San Antonio Texas
- TCAOM Propulsion Laboratory, Warren Michigan
- Lotus Engineering Ann Arbor, Michigan
- Automotive Testing and Development Services, Ontario California
- Ocean Air Environmental, Somis California

Fuels included in the plan are:

Pipeline grade natural gas (NG), Propane, Gasoline, Diesel (including DFM and bio diesel), and JP fuels. Variations in each of these fuels are proposed to reflect that fuels are not always in the same condition as when they were initially refined. Variations can result from:

- Les than optimum crude or feedstock, refining, processing, or storage
- Attack from oxygen and ozone

Bacteria growth

Fuel variation is a problem for the military, commercial, and individual users. The Army has the most challenging fuel problem, because of the wide range of operating temperatures, humidity, altitude, and fuel supply sources it encounters. Sub optimal fuels contribute to fuel system and injector fouling and are prone to less than complete combustion. When fuel burns partially in the combustion chamber less than optimal power and fuel economy are achieved. Partially burned fuel contributes to exhaust system fouling, smoke, emissions, maintenance, and stack fires

The plan, is to more fully develop a knowledge base in the area of low temperature hydrocarbon fuel reformulation and provide a more complete map of the best catalyst formula for each fuel.

Evaluations of APSI fuel catalysts will be performed on multiple fuel and combustion devices including

Simple combustion units:

- Lanterns
- Space heaters, burners, and furnaces

Reciprocating Engines fueled by Gasoline

- Small 2 and 4 stroke engines from fractional hp to 150 hp
- Large 4 stroke engines from 100 to 500 hp typical of the automotive fleet

Reciprocating Engines fueled by Diesel

- Small 25 hp to 75 hp utility and tractor engines
- Medium to Large 100 hp to 1000 hp 2 and 4 stroke gen-sets, land, and marine engines
- Very large 2 and 4 stroke diesel engines up to 5,000 hp.

Statement of Work / Statement of Objectives

Phase 1. Technical Objectives

The goals of the work effort to be conducted under APSI direction at UCONN are as follows:

- To produce next generation fuel catalysts (advanced fuel catalysts) that will be used for various fuels (CNG, gasoline, diesel, natural gas, propane) to improve combustion.
- To synthesize and characterize high surface area fuel catalysts.
- To study reactions of fuels in the presence of fuel catalysts.
- To provide the information needed to produce large-scale quantities of next generation fuel catalysts.

Technical Work Plan

This portion of the program would be performed at the University of Connecticut under direction of Dr. Al Berlin of APSI and Dr. Steven L. Suib of the University of Connecticut

Phase 1. Task 1. Catalyst Research & Formulation

APSI will extend the scope of the in-place APSI cooperative Research contract for pre-combustion catalysts with the University of Connecticut (UCONN) to create new catalyst formulations to promote constructive modification of various liquid and gaseous fuels including bio-diesel, diesel, JP-8, gasoline, natural gas, propane, and heating oil. The catalyst formulations shall include new chemical compositions and the deposition of catalysts on substrates to provide high surface area to weight (cm² / gram) ratios. This task also includes qualitative and quantitative laboratory testing at UCONN to assess the change in the fuels as a result of exposure to the catalyst.

Increasing The FFC Activity

Heterogeneous catalysts generally improve in activity when the surface area is increased. The existing APSI Fuel Catalyst is unusual in that it functions well as a catalyst at a surface area of about 81 cm² / 5 gal. liquid fuel. It has a surface area to weight ratio of about 1.7 cm² / gram. Another heterogeneous catalyst, for example, the catalytic converter has a surface area of about 85 million cm²/gram. Hence, one of the means at our disposal to improve the activity of the FFC is to increase its surface area.

Another way of improving the activity is to add materials or elements that increase the activity synergistically. These are often given names such "promoters." Two promoters that may improve the activity of FFC are the metals Cerium and Lanthanum.

Several methods of improving the activity, by increasing the surface area of FFC, are contemplated. One method that will be explored is to absorb the FFC metals on to beta-aluminum oxide substrate. This type of aluminum oxide has a surface area of about 350 square meters per gram. Polymeric support systems will be designed to incorporate FFC powders.

The effects of increased surface area and promoters will be studied by analyzing the off- gases from the interaction of the fuel catalyst under development and various fuels by GC/Mass Spec, and by analyzing compositional changes induced in whole fuels and model compounds of fuels by NMR / PMR.

Studies are proposed to probe methods of producing more active, more durable, more microbe toxic, and possibly less expensive Fitch Fuel Catalysts (FFCs). We will study surfaces of the catalyst to increase the surface area and create unusual new supporting materials for the catalyst. We also intend to examine the synergistic effects of magnetic and electrical fields with FFCs on various types of liquid and gaseous fuels.

An Experimental Program that has been designed to accomplish these objectives. It employs a mathematical and statistical approach to determine important variables in these scientific studies. The activities the Experimental Program will cove are:

1. Design Novel Fitch Fuel Catalysts (FFC).

Nanoparticle FFC's plated on supports (a) Measure activity on fuels (b) Surface Area versus Activity.

2. Characterization of Surfaces of the Fitch Fuel Catalyst. Atomic force microscopy and scanning tunneling microscopy studies of FFC. Effect of Surface Area, BET and pore size distribution on Activity of FFC.

3. Generation and Characterization of Acid Sites in Next Generation Fitch Fuel Catalysts.

FFC's supported on Solid Acids (a) Brönsted acids (b) Lewis Acids

4. Use of Supports for Fitch Fuel Catalyst. Nanoparticle FFC's adsorbed on Supports (a) Alumina (b) MnO₂ paper.

- 5. Degradation and Control of Bacteria in the presence of Fitch Fuel Catalysts.
- 6. Effects of Magnetic, Electronic, and Electromagnetic fields on Fuels in the presence of the Fitch Fuel Catalyst. MW heating, MW plasmas in FFC applications of fuels.
- 7. Chemical Vapor Deposition (CVD) and Plasma Assisted CVD Methods for Preparation of Fitch Fuel Catalysts. Preparation of materials via deposition techniques.

Phase 1. Task 2. Catalyst Scale Up and Verification We will take the most positive results of Task 1 and:

- Develop manufacturing sources to manufacture selected new catalyst formulations in pilot lot quantities.
- Conduct standardized ASTM (or equivalent) tests too further corroborate the laboratory tests performed in Task 1.

Catalyst Laboratory Verification

We will conduct accelerated tests to assure the selected catalysts scaled up for combustion testing are stable for long periods of time and do not react negatively with hydrocarbon fuels nor corrode. Using the same techniques employed to measure effectiveness of the novel catalysts we will confirm that scaled up catalyst are capable of inducing the changes in the various HC fuels that have been detected and determined in Task 1.

Phase 2. Technical Objectives

Evaluate the catalysts developed and verified in Phase 1 to actual and / or simulated operation of internal combustion engines, fuel injectors, combustion burners, furnaces, lanterns and other hydrocarbon fueled devices. Compare the conversion efficiency, fouling characteristics, and exhaust emissions signature of the combustion device with and without the application of fuel catalysts.

Phase 2. Technical Work Plan

Fuel Injector Flow Testing

1. Fit APSI Fuel Catalyst to fuel injector flow test rig (Southwest Research Institute) with fuels known to induce fouling, measuring effectiveness of the fuel catalyst(s) to mitigate fouling.

Combustion Testing

- 1. Capture Baseline information for the combustion unit with the commercially available fuel
- 2. Fit the APSI Fuel Catalyst to combustion device.
- 3. Measure changes in the devices performance compared to baseline.
- 4. Optimize efficiency of each combustion device in the presence of the fuel catalyst

Data recorded during combustion equipment tests:

- a) Fuel consumption
- b) Exhaust gas composition
- c) Yield (heat, light, or shaft power (useful work) produced) per unit of fuel.
- d) All other operating parameters of the equipment in test

The proposed testing schedule for the Demonstration of Benefits will be accomplished during month 7 through 11 (See Schedule)

Phase 2. Planning, Reporting and Administration

- Prepare detail plans for each task within 30 days of the initiation of Phase 1 and. Maintain and amend these plans in coordination with the Government Technical Manager for the project.
- Prepare quarterly technical reports, monthly financial reports and a final technical report for the project.
- Prepare and present the results of the project work at roughly the contract mid-point and after the completion of the technical work of the project.
- Communicate significant technical and project results or issues by telephone or email to the Government Technical Manager at the earliest possible time after the event occurs.

Table of Project Milestones

		rable of Project Milestones			
Task	Milestone	Title	Project	Internal	Deliverable
#	#		Month	Project	То
				Deliverable	TACOM
1		FORMULATE NEW CATALYSTS			
	1	New Catalyst Formulation for Task 2	3	X	
	2	As above	9	X	
	3	As above	12	X	
2		CATALYST SCALE-UP			
	4	Deliver Catalyst Pilot Quantity for Task 3	6	X	
	5	As above	11	X	
	6	As above	13	X	
3		DEMONSTRATION OF BENEFITS			
	7	Start Lantern Demonstrations	7	X	
	8	Start Injector Demonstrations	7	X	
	9	Start Furnace/Burner Demonstrations	12	X	
	10	Start Engine Demonstrations	15	X	
	11	Complete all Demonstrations	18	X	
4		PLANS, REPORTS,			
		ADMINISTRATION			
	12	Update Project Plan from Proposal	1		X
	Not on	Technical Report	3,6,9,1		Х
	Gantt	·	2,15		
	Not on	Financial Report	Each		X
	Gantt	·	Month		
	13	Presentation #1 at TACOM	8		X
	14	Presentation #2 at TACOM	17		X
	15	Final Technical Report	18		X

Project Gantt Chart

